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USE OF THE HOT GAS SLURRY TECHNIQUE FOR SOLID SAMPLE INTRODUCTION FOR ICP-AES.

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INTRODUCTION

One of the primary limitations of conventional laboratory instrumentation is its inability to do very low level determinations directly in solid materials. Recent experience in our laboratory with "real solid samples" has led us to investigate slurry sample introduction methods for ICP-AES. Slurry methods have the distinct advantage that it produces a static signal allowing sufficient time for background correction methods and even rapid scanning spectrometers to be used. Analysis by the slurry methods are simple to perform and require very limited modification to the ICP system normally used for liquids. Our initial experiments demonstrated to us that slurries did offer many of the claimed advantages, e.g. calibration with aqueous standards, however detection limits were significantly reduced. This is to be expected since a 1% slurry is, in essence, a 1:100 dilution of the sample. Higher percentage slurries could be used but handling of the suspended solid becomes more difficult as the load increases.

In our search for better detection limits with slurries we have followed a general methodology used by Veillon and Margoshes[1]. The use of a heated spray chamber followed by a condenser was reported to result in a dry aerosol. Configurations of this type are now common with ultrasonic nebulisers where

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Veillon, C.; Margoshes, M. *Spectrochim. Acta* **1968**, 23B, 553-555.

the very large amount of solvent must be removed before reaching the atomisation source. This papers reports on the use of this general design with a Babington type nebuliser for the introduction of slurry. The key addition to the system is the use of a hot gas through the nebuliser. This new system was expected to provide a sufficient input of thermal energy through the nebuliser gas and the walls of the spray chamber for the development of either smaller droplets or possibly aerosol particles which should transport to the plasma with much higher efficiency. Higher transport efficiency should result in improved detection limits if plasma excitation conditions remain essentially unchanged.

EXPERIMENTAL

HOT NEBULISER GAS SLURRY SYSTEM

A Legere nebuliser[2] and spray chamber were used with a cooled inner coil condenser similar to the arrangement of Veillon and Margoshes[1]. The normal PVC sample input tube was replaced by a stainless steel tube to allow the use of a high temperature environment. The sample is fed to the nebuliser by a peristaltic pump. The argon gas was heated to the desired temperature when passed through copper tubing wrapped with a heating tape. The temperature of the gas was adjusted with an electronic temperature controller which receives feedback from a thermocouple placed in the copper gas line immediately before the nebuliser body. The glass spray chamber was wrapped with a heating tape which extended from the nebuliser to over the glass connector leading to the condenser. A variable power supply was used to control heating of the spray chamber while the surface temperature was measured with a thermocouple. The cooling water for the condenser was maintained at 10 C (+/- 2 C) by circulation in an ice-water bath. The inner coil condenser was operated in a vertical position. The spray chamber is at an 8 degree angle with the horizon to allow free flow to the drain. In this work, the heated spray chamber was used to maintain the hot environment produced by the hot nebuliser gas along the path of the aerosol to the condenser.

SAMPLE PREPARATION

The composition of the aqueous sample was of 20 ppm in aluminum, cadmium, copper, lead, nickel and-zinc. Analytical grade salts or spectroscopic grade powders were used for the preparation of those solutions. The marine sediment reference material BCSS-1 (National Research Council of Canada) was used for the slurry sample. BCSS-1 is delivered in a format which passes No 120 (125 micron) screen. Particles of less than 3 microns (97 % of the particles) were obtained with a thirty minutes grinding in an agate vial. The 0.5% (w/v) BCSS-1 suspension in water was treated with a 30 minutes sonication using an ultrasonic bath and then magnetically stirred during the analysis to keep the sample uniformly suspended. All samples were made with deionized doubly-distilled water.

PROCEDURE

A Thermo Jarrell-Ash ICAP 61 direct reading spectrometer was used for all measurements with conventional operating conditions. Intensity measurements were obtained for a water blank, the multielement aqueous solution and the 0.5% BCSS-1 slurry. The signals used were the average of 10 repeats of a 10 seconds integration. For both types of samples, aluminum, cadmium, copper, nickel, lead and zinc were monitored. With the exception of

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Legere, G.; Burgener, P. *ICP Info.Newsl.* **1985**, 11(7), 447-456.

aluminum, all are part of the trace constituent list for BCSS-1, our target "real sample".

An optimisation of the hot nebuliser gas sample introduction system was done for two factors: (1) the temperature of the nebuliser gas and (2) the temperature of the spray chamber. The gas temperature effect was studied over the range of 23 C to 235 C. Spray chamber temperatures ranged from 23 C to 250 C for the liquids and up to 185 C for the slurry.

RESULTS AND DISCUSSION

The Hot Nebuliser Gas Slurry system was first tested and optimised for liquid samples. An optimum Es/Blk (Emission signal to blank) was found at a gas temperature of 205 C and a spray chamber temperature of 110 C. The improvement of the optimisation factor at a specific operating condition was calculated with respect to the optimisation factor found when using the same system at room temperature. For those liquid analysis conditions, an improvement of 4.6 of the Es/Blk was obtained.

The optimisation for slurries showed slightly different optimum conditions than those for liquids. An input gas temperature of 235 C instead of 205 C with the same spray chamber temperature (110 C) was the most efficient. Under these conditions, an Es/Blk improvement of 6.5 was obtained. Running the BCSS-1 slurry under the liquid sample optimum conditions, an improvement of 4.9 was observed.

The use of the hot nebuliser gas system for the nebulisation of either liquids or suspensions results in a significant improvement of the emission signal-to-blank-ratio observed. Therefore the analytical performance of the ICP-AES system, particularly the detection limit, could be improved by this sample introduction system. In its current state, a short term instability of the signal is observed which results in poor noise levels. Work is in progress to solve this problem.

At this point we have optimised the temperatures of the spray chamber, gas heating system and the viewing height for one set of conditions. To gain a more correct estimate of the power of the system, a global optimisation would be necessary. Effect of the operating conditions such as nebuliser gas flow, nebuliser liquid flow, plasma power and gas flows and condenser would have to be studied. While we are encouraged by the improvement of 6.5, we expect that further improvement should be possible when these other factors are included in the optimisation process.

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